MICROSTRUCTURAL STUDIES ON ELECTRON IRRADIATED POLYMERS

A synopsis of the thesis to be submitted to the Mangalore University for the award of the degree of Doctor of Philosophy in Physics

Submitted by

ISMAYIL

Under the Supervision of

Dr. V. RAVINDRACHARY

Department of Studies in Physics
Mangalore University,
Mangalagangotri - 574 199, India

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Candidate	ISMAYIL Research Scholar Department of Physics, Mangalore University, Mangalagangotri – 574 199
Research Guide	Dr. V. Ravindrachary Associate Professor Department of Physics, Mangalore University, Mangalagangotri – 574 199
Work place	Department of Physics, Mangalore University, Mangalagangotri – 574 199
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Synopsis of the PhD Thesis

Nowadays the polymers play a very important role in all walks of human life due to their high utility, processibility, synthetic flexibility and other physical properties, hence the present era is known as ‘polymer age’. Some of the technological applications of polymers comprises of optics, electronics, biotechnology, photonics, space research etc. It is known that the physical and chemical properties of a polymer required for a specific application may be obtained by doping and/or irradiation techniques. Generally the microstructural modification in polymers is a key instrument for the design of new products and processes for different applications. The irradiation of polymers with ionizing radiations such as electron beam, heavy ion etc. significantly alters the microstructural as well as the macroscopic properties of the polymer [1-3] and is known to be an established tool to tune the properties of a polymer. Hence the studies on irradiated polymeric materials are of rapidly increasing area of research interest.

The major chemical changes that takes place within polymers due to irradiation with ionizing radiation are; gas evolution (H_2, CO_2 etc), creation of new double bonds, formation of free radicals and vacancy clusters, main chain-scission (C–C bond scission), radical–radical recombination (cross-linking) etc. [4-6]. Among these chemical changes, cross-linking and chain scission are the most general types of reactions in irradiated polymers. Generally, the cross-linking process leads to the formation of chemical bonds between two adjacent polymer molecules and this reaction increases the molecular weight of the polymer until the material is bound into an insoluble three-dimensional network. On the other hand, chain scission or fracture of polymer molecules decreases the molecular weight and increases the solubility. Both these reactions can

significantly alter the physical properties of a polymer [4]. However, the degree and direction of change are not the same for all polymers.

It is known that the exact reaction process, reaction rate and resultant products due to irradiation are different for different polymers. They depend on the molecular structure, chemical environment and exposure dose. Parameters such as morphology, chemical structure of the polymer, dose and irradiation media determine whether cross-linking or chain scission is the dominating effect upon irradiation [7-9]. The degree of these modifications depends on the structure of the polymer and the conditions of treatment before, during and after irradiation. Hence thorough control of all of these factors facilitates the modification of polymers by radiation processing. Further the modifications in the microstructural properties of the polymer due to irradiation depend on the irradiation dose and the way in which reaction species interact with the host matrix. Hence the understanding and processing of polymer with different irradiation dose is a field of continuing research.

As seen earlier, polymers generally undergo structural changes under irradiation and its utilization for industrial applications requires a basic understanding of their material properties. A key issue in this regard is the relationship between the macroscopic properties of the polymers with the atomic-scale free volume related microstructure and their changes due to processing. Under irradiation, the physico-chemical changes occurred within the polymer via free radical formation and crosslinking are thought to modify the cavities or free volume holes of atomic and molecular dimensions that appear in amorphous polymers. From various study it is clear that the free volume change within the polymer greatly affects structural, electrical and optical properties of polymers [10-11]. Although the concept of free volume in polymers has been known for nearly half a century, there are only limited experimental data available in literature. This is mainly due to a lack of suitable probes for open volumes of molecular dimensions.

In recent years, the positron annihilation is a well-established technique for studying subatomic size free volumes in polymers. When an energetic

positron from a radioactive source enters a condensed medium like a polymer, it thermalizes by losing its energy in a very short time. Then it annihilates with an electron of the medium by a free annihilation process, or the positron is trapped in a defect site and annihilates with an electron of the defect site, or forms a bound state called the positronium (Ps). Usually the Ps exist in two spin states, a para positronium (p-Ps, particle spins antiparallel) which annihilates with a lifetime of 0.123ns and an ortho positronium (o-Ps, particle spins parallel) which decays with a lifetime of 142ns in vacuum. In polymers, the lifetime of o-Ps is reduced to few ns due to the pick-off process. As a result of these, a positron annihilation lifetime spectrum in a polymer shows different components. Particularly, o-Ps is important one in polymeric research, because the o-Ps lifetime can be quantitatively correlated with the size of free volume and its intensity can be correlated with the number of free volume, based on the free volume model [12-13]. Hence both lifetimes and intensities are sensitive to the physico-chemical environment of the sites of annihilation.

The optical property of polymers is of great importance in device applications. The modification of these properties justify the need for studying the effects of irradiation on the optical properties of polymers including absorption, band gap and fluorescence behaviour. Among the optical polymers, Poly(vinyl alcohol) (PVA) and Poly(methyl methacrylate) (PMMA) are the important polymers used for different applications due to their good physical properties [3-5]. Recent studies have shown that in various metal ion doped PVA films, the chemical interaction of dopant and polymer groups affects polymeric microstructural properties and further modifications can be done using irradiation. Hence there is a lot of scope for improving the optical properties such as fluorescence and electrical properties of the polymer by irradiation and is the immense filed of research. Since the fluorescence and electrical properties of the polymer are mainly depends on the free volume content of the polymer matrix, a free volume related microstructural study and its correlation with the above mentioned properties is the interesting topic for current research.

Aim and Objectives of the Present Study

In view of the above, the present investigation aims to understand the effect of 8MeV electron beam irradiation on the free volume related microstructure and hence the physical properties of the polymer matrix. In this context two commercially available polymer hosts such as an amorphous ploy(methyl methacrylate) (PMMA) and a semicrystalline poly(vinyl alcohol) (PVA) have been selected. Sodium bromide (NaBr), Lithium tetra borate ($Li_2B_4O_7$) and an organic NLO compound (a chalcone derivative) 1-(4-methylphenyl)-3- (4 - N, N- dimethylaminophenyl) - 2-propen-1-one (MPDMAPP) have been used as dopant for these polymers.

It is known that property of the doped polymer depends on the chemical nature of the dopant and the way in which it interact with the host polymer. Here two metal salts were selected as dopants to understand the degree of complexation and its behavior on the conductivity of the polymer. Similarly the doping of organic chalcone will induce the fluorescence property to the polymers. These induced fluorescence property depends upon the free volume nature and the morphology of the polymer. Further, the physical properties like conductivity and optical property of the polymeric material can be further modified for particular application using electron beam irradiation. The pure and doped polymers are irradiated with 8MeV electron beam using the Microtron, an electron accelerator. The modifications occurring within the polymer due to electron irradiation and the induced microstructural changes in doped polymer composites are studied by different experimental techniques. The free volume related microstructural changes are studied using Positron Annihilation Lifetime Spectroscopy (PALS) complemented with X-ray Diffraction (XRD) technique. The modified chemical and optical properties are studied using FTIR, UV-Visible spectroscopy and Steady State Fluorescence Spectroscopy. The thermal properties are studied using Thermogravometric Analysis (TGA) and Differential Thermal Analysis (DTA). All these extensive studies are presented in **eight chapters** of the thesis.

Chapter I provides detailed ***introduction*** to polymers, doping of polymers and effect of electron irradiation on different polymers including the literature survey on the current status of irradiated polymers. It also gives the basic idea about positron annihilation and other complimentary characterization methods including the theoretical aspects of free volume and positron annihilation as well as other complementary techniques in the mentioned topics. The motivation, aims and objectives for the present investigation are also presented.

Chapter II describes the ***experimental and characterization techniques.*** The sample preparation, electron irradiation of the polymers using the Microtron and experimental characterization techniques like UV-Visible spectroscopy, FTIR, X-ray Diffraction (XRD), Thermogravimetric Analysis (TGA), Differential Thermal Analysis (DTA), Fluorescence Spectroscopy and Positron Annihilation Lifetime Spectroscopy (PALS) have been discussed. The experimental details like data accumulation and the analysis of positron lifetime spectra are described extensively.

Chapter III deals with the ***optical and microstructural studies on electron beam irradiated Poly(methyl methacrylate)***. In this study the standard amorphous polymer Poly(methyl methacrylate) (PMMA) has been used as sample and is irradiated for different electron doses. The modifications in the chemical, optical and microstructural properties of electron irradiated PMMA films have been investigated using different techniques. The FTIR result indicates that the irradiation causes chain scission and creates C=C bond within PMMA, which in turn affect the optical properties of the film. The observed results understood by invoking the formation of carbonaceous clusters within the polymer composite upon irradiation. This study shows that the irradiation process enriches the carbonaceous clusters and vacancy type defects which increase with irradiation dose. These increase in carbonaceous clusters affects the molecular mobility and results in the increase in free volume size within the electron irradiated PMMA. The XRD studies indicates that electron beam irradiation on PMMA films increases the degree of amorphousness of the

sample. From the PALS study, it is observed that, the o-Ps lifetime τ_3 and free volume size V_f, remains steady up to 100kGy and then it increases smoothly for higher doses. On the other hand o-Ps intensity I_3 varies in three steps: First I_3 decreases smoothly up to 100kGy and then increases up to 200kGy and after 300kGy it remains constant with increasing dose. These results can be interpreted based on the free volume model. The observed smooth decrease in o-Ps intensity, up to 100kGy is attributed to the presence of low amounts of chemically active species (free radicals) which are produced by irradiation. Further, the observed increase in τ_3 & I_3 after 100kGy seems to fit quite well with the interpretation based on the process of chain scission as observed in FTIR studies. It is observed in FTIR studies that the electron irradiation causes chain scission results in enhanced bond cleavages and H_2 gas evolution which leads to the formation of sp^2 sites within PMMA. Generally, the π bonding in sp^2 sites favors the pairing, so that these sp^2 sites in the polymer matrix tend to organize into carbon clusters and may act as positron scattering centers within the matrix. These scattered positrons from the centers may trap in the 'vacancy like' defects within the polymer matrix. In the presence of these positron trapping centers or positron scattering centers, the recombination of positron-electron pairs with small separations take place as a result the positron mobility reduces. This recombination process within the amorphous matrix may leads to an increase in the positronium formation. This feature also reflected in enhancement of interchain distance (observed in XRD studies) as well as flexibility of the polymer film and in turn results in the increase of free volume size and its concentration.

Accordingly PMMA suffers main chain scission upon irradiation, low molecular fragments are produced which may "plasticize" the polymer and the accumulated gases may then convert the whole specimen into a "foamed" structure. This may cause an increase in the molecular mobility and hence decrease in optical activation energy (E_a) as well as increase in the free volume size in PMMA. The variation of I_3 with E_g suggests that the probability of Ps formation in polymer may be well correlated with the sp^2 content; the higher the

sp^2 content, the lower the Ps formation. In this study a correlation between positron lifetime and optical results are obtained and it shows the usefulness of PALS measurements in understanding the free volume related microstructural modifications within the irradiated PMMA.

The ***free volume related fluorescent behaviour in electron irradiated Chalcone doped PVA*** are described in **Chapter IV.** This work is carried out to tune the fluorescence and structural properties of the chalcone chromophore 1-(4-methyl phenyl)-3-(4-N,N, dimethyl amino phenyl)-2-propen-1-one (MPDMAPP) doped PVA polymer composite by electron beam irradiation. The FTIR spectral study on the irradiated film suggests that the crosslinking of the polymer chains takes place within the matrix upon irradiation. Using UV-Visible absorption spectra the optical parameters like optical energy bandgap and activation energy were determined. The variation of these parameters suggests the existence of defects and molecular ordering within the irradiated sample, which leads to the fluorescence enhancement in irradiated PVA/MPDMAPP films. The variation of fluorescence emission intensity indicates that the fluorescence enhancement occurred more efficiently under irradiation conditions due to the crosslinking of polymer chains.

The PALS studies reveals that the o-Ps lifetime τ_3 and free volume size V_f remains almost constant upto 150kGy and decreases gradually for higher doses. The intensity I_3 and fractional free volume slightly decreases with irradiation dose. The decrease in τ_3 with increasing electron dose after 150kGy is due to the intermolecular crosslinking of polymer chains by electron beam irradiation. These results indicates that the electron irradiation produce free radical as well as cross-linking which changes the characteristics of polymers. The decrease in τ_3 is understood by invoking the change in the free volume brought about the formation of new bonds or cross-linking. In the cross-linked state, the polymer chains are expected to have a closer packing, hence a reduction in free volume is observed at this stage. On the other hand, the decrease in I_3, which is associated with a decrease in the number of free volume holes, seems to fit quite well with

the interpretation based on the process of cross-linking. This reduction in I_3 with dose is considered to be due to the structure change. Thus, the Positron annihilation result shows that electron irradiation decreases the free volume after certain irradiation dose as a result the chalcone chromophore molecular rotation has been hindered. Hence under this restricted condition the chromophore molecules likely to emit enhanced fluorescence on excitation. Thus, the mobility of a fluorescent probe is directly related to the free volume around it. Hence the electron beam treatment studies helps to tune and to investigate the improvements in the microstructural properties of Chalcone doped Poly(vinyl alcohol) films.

Chapter V presents the ***free volume related fluorescence properties of electron irradiated chalcone doped PMMA films*.** In this study the effect of electron irradiation on the fluorescence and other microstructural properties of PMMA/MPDMAPP composite films are carried out. Here an attempt has been made to understand the microstructural modification of fluorescent films with thermoplastic amorphous PMMA as a host matrix. The FTIR spectral study on the irradiated films suggests that the crosslinking of the polymer composite chains takes place within the matrix upon irradiation. Using UV-Visible absorption spectra, the optical parameters like optical energy bandgap and activation energy were determined. The variation of these parameters suggests the existence of defects and molecular orderness within the irradiated sample, which leads to fluorescence enhancement in irradiated PMMA/MPDMAPP films. From the fluorescence studies, the increase in the intensity of fluorescence emission indicates that the fluorescence enhancement occurred more efficiently under irradiation conditions due to the crosslinking of the polymer chains.

The PALS study depicts that the o-Ps lifetime τ_3 as well as free volume size V_f decreases with irradiation dose initially upto 150kGy and increases for higher doses. On the other hand, the o-Ps intensity I_3 and fractional free volume ($V_f I_3$) decreases with irradiation dose. This variation is similar to that of interchain distance observed from XRD studies. From the variations of τ_3 and I_3, the information on the microstructural changes in amorphous regions can be

obtained. The variation in τ_3 suggests that the crosslinking of polymer chain at lower doses decreases the free volume size and at higher doses the process of chain scission increases the free volume size, as observed from FTIR and XRD studies. The decrease in I_3 indicates that the free volume density decreases with irradiation dose. Thus, the Positron annihilation result shows that electron irradiation decreases the free volume after certain dose and hence the chalcone chromophore molecular rotation has been hindered. Hence under this restricted condition the chromophore molecules likely to emit enhanced fluorescence on excitation. Thus, the mobility of a fluorescent probe is directly related to the free volume around it. It is found that the chalcone molecules in the crystalline state are less fluorescent but are strongly fluorescent within the polymeric matrix. Hence the incorporation of such multifunctional photochromic chalcone derivative in polymers exhibits the photo-induced electron transfer reaction, which may be utilized for fast response time and other photonic devices application. It is observed that, in the case of PVA/MPDMAPP, crosslinking is the main process whereas in the case of PMMA/MPDMAPP, both crosslinking followed by chain scission processes responsible for the microstructural modifications of the polymer matrix.

In **Chapter VI** the ***electron beam irradiation effects on the microstructure of PVA:NaBr polymer composite*** has been studied extensively. This work aims to understand the effect of electron beam irradiation on the free volume related microstructure and its influence on electrical conductivity of PVA:NaBr composite at room temperature. The effect of irradiation on microstructure and conductivity of PVA-NaBr polymer composite films were investigated using different techniques. The results reveal that the cross-linking and the onset of chain scissions within the polymer matrix due to irradiation affects the free volume content and hence the electrical properties of the polymer. From the PALS studies it is observed that the third lifetime component τ_3 as well as free volume size decreases drastically upto 150kGy and afterwards it increases with electron dose. The corresponding o-Ps intensity I_3 and fractional free volume behaves in similar fashion as that of τ_3. It may be suggested that the

decrease in τ_3 is due to the change in the free volume brought about by the formation of new bonds or 'cross-linking'. On further increase of dose up to 300kGy, the onset of chain scission process seems to be dominated over cross-linking as a result the free volume size and intensity increases. Moreover, the most efficient process induced by irradiation at higher doses is the production of the free radicals, ions or carbonaceous clusters. The formed free radicals may recombine in various ways; promoting important chemical and structural changes within the polymer matrix. The presence of such free radicals will contribute to the enhancement of conductivity of the PVA:NaBr polymer composites. Further, the FTIR results show that the irradiation creates carbonyl groups and possibly some double bonds which in turn contribute to the enhancement of conductivity. The observed enhanced conductivity due to irradiation also suggests that the double bond was expected to be formed in very low amounts, which is insufficient to reduce positronium formation at low dose and an increase in positronium formation at higher dose. Hence an increase in I_3 at higher doses is expected in PVA:NaBr polymer electrolytes.

In the present system the PVA/NaBr interaction increases the mobility of ions, resulting in an increase in ionic conductivity and is directly related to change in free volume within the irradiated composite. Here it can be suggested that the crosslinking network provides hopping sites for Na^+ ions in the polymer matrix and this enhanced networking leads to increase in the ionic conductivity of the films. At lower irradiation doses upto 150kGy, the τ_3 or V_f values reduces significantly and such a decrease is attributed to coordination of the Na^+ ions with solvating sites in the polymer. Hence, the change in free volume within the polymer affect the mobility of ions and polymer segments and hence the conductivity. Thus, at lower doses, crosslinking network provides hopping sites for ions and hence hopping mechanism is responsible for enhancement of conductivity. At higher doses, the free volume size increases due to chain scission, consequently segmental motion causes the ionic transport and results in conductivity enhancement.

Chapter VII highlights the ***electron beam induced modifications in the microstructure of Lithium Tetra Borate doped Poly(vinyl alcohol)*****.** The $Li_2B_4O_7$ doped PVA films were irradiated with 8MeV electron beam and the induced modifications in optical and electrical properties were investigated. In this chapter an attempt has been made to understand the enhancement of conductivity of PVA in the presence of $Li_2B_4O_7$. The FTIR result indicates that the irradiation causes crosslinking within $Li_2B_4O_7$ doped PVA, which in turn affect the optical properties of the film. The UV-Visible study reveals that a new absorption peak emerges at 280nm under electron beam irradiation. In this case two optical energy band gaps and activation energies were observed after electron irradiation. This may be attributed to the formation of additional energy levels due to the creation of defects and carbonaceous clusters upon irradiation. The variation of the energy band gap upon irradiation can be attributed to the increase in the conductivity of the composite films. This increase in conductivity upon irradiation results were interpreted by invoking the motion of the ion. Here the polar groups of polymer interact with ions and are linked covalently to each other. The moving polymer segment with associated carrier ions is much larger than the volume of naked Li^+ ions and the polymer segmental motion occurs only when the holes having a value greater than some critical value (approximately equal to the size of one polymer segment unit with associated carrier ion Li^+). With the increase of electron dose, free volume size increases, and that facilitates the micro-Brownian motion of main chain. That is the motion of polymer segments with Li^+ will be speeded up. So the polymeric segmental motion with associated carrier ions appears to be the rate-determining step for ionic conduction. The PALS study suggests that the segmental motion with associated carrier ions causes the ionic transport, and the ionic transport strongly depends on the probability of redistribution of the free volume, which is large enough to allow ionic migration. It is observed that, in the case of PVA: $Li_2B_4O_7$ chain scission is the main process whereas in the case of PVA:NaBr both crosslinking followed by chain scission processes responsible for the microstructural modifications of the polymer matrix.

Finally in **Chapter VIII** the overall summary of the present investigations and the scope for further study are discussed. It is understood that the fluorescence enhancement is more in case of semicrystalline PVA rather than amorphous PMMA matrix, as the hindrances of the fluorescent probe molecules within polymer free volume is the main reason for fluorescence enhancement. It is observed that the doping of metal salt NaBr in to the PVA will modify the microstructure and enhance the electrical conductivity of the semicrystalline PVA. Similarly the Lithium tetra borate salt will contribute to the enhancement of conductivity of the polymer composite. The results of these investigations are published in refereed journals and some are presented in the national and international conferences / symposia. They are given in the form of list of publications.

Mangalagangotri (Ismayil)

Date: 28-01-2012 Research Student

(Dr. V. Ravindrachary)

Research Supervisor

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